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Procedia Engineering 56 (2013) 619 – 624
Procedia Engineering
www.elsevier.com/locate/procedia

5th BSME International Conference on Thermal Engineering

Optimisation of Bio-oil Extraction Process from Beauty Leaf (*Calophyllum inophyllum)* Oil Seed as a Second Generation Biodiesel Source

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Abstract

The Beauty Leaf tree *(Calophyllum inophyllum)* is a potential source of non-edible vegetable oil for producing future generation biodiesel because of its ability to grow in a wide range of climate conditions, easy cultivation, high fruit production rate, and the high oil content in the seed. This plant naturally occurs in the coastal areas of Queensland and the Northern Territory in Australia, and is also widespread in south-east Asia, India and Sri Lanka. Although Beauty Leaf is traditionally used as a source of timber and orientation plant, its potential as a source of second generation biodiesel is yet to be exploited. In this study, the extraction process from the Beauty Leaf oil seed has been optimised in terms of seed preparation, moisture content and oil extraction methods. The two methods that have been considered to extract oil from the seed kernel are mechanical oil extraction using an electric powered screw press, and chemical oil extraction using n-hexane as an oil solvent. The study found that seed preparation has a significant impact on oil yields, especially in the screw press extraction method. Kernels prepared to 15% moisture content provided the highest oil yields for both extraction methods. Mechanical extraction using the screw press can produce oil from correctly prepared product at a low cost, however overall this method is ineffective with relatively low oil yields. Chemical extraction was found to be a very effective method for oil extraction for its consistence performance and high oil yield, but cost of production was relatively higher due to the high cost of solvent. However, a solvent recycle system can be implemented to reduce the production cost of Beauty Leaf biodiesel. The findings of this study are expected to serve as the basis from which industrial scale biodiesel production from Beauty Leaf can be made.

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Keywords: Beauty Leaf; second generation biodiesel; bio oil extraction

1. Introduction

Demand for energy is increasing every day due to the rapid growth of population and urbanisation. Biomass is emerging as one of the promising environmentally friendly renewable energy options if the major conventional energy sources like petroleum oil, coal and gas become depleted. Biomass can be converted into liquid and gaseous fuels through thermo-chemical and biological methods. Fuels produced from these technologies are referred to as biofuels. It is generally held that biofuels offer many benefits over conventional petroleum fuels, including availability from locally available biomass sources, reduction of greenhouse gas emission, biodegradability, and contributing to sustainability [1]. However, biofuels

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1877-7058
Selection and peer review under responsibility of the Bangladesh Society of Mechanical Engineers
doi:10.1016/j.proeng.2013.03.168

contain oxygen levels of 10–45% by mass while petroleum has essentially none. This makes the chemical properties of biofuel more favorable for complete combustion. In addition, biofuels from all sources have very low sulphur content and many have a low nitrogen level which make them more eco-friendly [2]. As a consequence, biodiesel is widely used as an alternative fuel for diesel engines, whereas ethanol is used to replace gasoline [3]. In general, biodiesels are fuel obtained through the esterification of oil derived from plants or animal fat. While the biodiesel properties are comparable to regular petrodiesel, the primary difference is they are not derived from petroleum sources such as crude oil.

Currently, the sources of biodiesel under investigation include soybean oil [4], sunflower oil, corn oil, used fried oil, olive oil, rapeseed oil [5], castor oil, lesquerella oil, milkweed seed oil [6], Jatropha curcas, Pongamia glabra (karanja), Madhuca indica (mahua) [7] and palm oil [8]. These are usually produced from edible oil feedstock and are known as first generation biodiesels [9]. The most contentious issue affecting the production of first generation biodiesel is the use of agricultural land for biodiesel production. This issue is commonly referred to as the "Food verses Fuel" debate, in which the main two issues are the use of edible crops for biodiesel production, and the amount of land space devoted to growing inedible crops. Therefore an alternative must be considered which eliminates the disadvantages of first generation biodiesels. Research is currently taking place on second generation biodiesels which are targeted at addressing the "Food verses Fuel" debate [10]. However, the current production of the above mentioned feedstock does not come close to a value representative of replacing fossil fuel use. This is more prevalent when land use and potential yields are considered, which eventually affects the feasibility of biodiesel production on an industrial scale. In a recent study [11], a large number of native species have been assessed for growth on degraded land in Australia which produces biodiesel at appreciable quantities. Among them, Beauty Leaf (*Calophyllum inophyllum*) has been identified as the most suitable feedstock for future generation biodiesel feedstock. However, its potential as a source of second generation biodiesel is yet to be assessed due to a lack of knowledge of the production process. This study aims to describe the optimised oil extraction process from Beauty Leaf seeds in terms of seed preparation, moisture content and oil extraction methods.

2. Beauty Leaf (Calophyllum Inophyllum) plant

Calophyllum inophyllum, or more commonly known as Beauty Leaf, is a moderate sized tree that grows between 8–20 m tall and is most notable for its decorative leaves and fragrant flowers, as can be seen in Fig 1. The tree grows in tropical and sub-tropical climates (typical temperature range of 18–33^{o}C) close to sea level. Beauty Leaf trees grow in free draining soils near shorelines; however, it has been seen to grow in various different clay soils both within Australia (Fig.1) and various parts of southern and more central Asia such as Sri Lanka and India [12]. It is a moderately quick growing tree that can grow up to 1 m tall within a year. It has also been seen to flourish even with the presence of weeds and other species, so the plant can be grown in mixed cultures and weeding is not necessary [13]. The tree bears fruit twice a year and a healthy tree produces around 8,000 fruits which contain a kernel within a hard husk. The fruits can be harvested either directly from the tree once they are fully matured or once they have fallen off. As it has been noted that a number of animals eat the fruit, it is a safe practice to harvest the fruits from trees just before they fully mature. The kernels themselves are contained in a hard shell, as seen in Figure 1. The kernels extracted from the fruit have a dry weight of about 5 g. With around 4,000 fruits per harvest (or 8,000 per year, as fruits are produced twice a year), this results in around 40 kg of kernels per tree per year. The spacing of the trees should be 5 x 5 m, allowing 400 trees per hectare. With 400 trees per hectare, a total of 16,000 kg of kernels can be produced per year. These kernels contain about 25–60% useful oil on a unit mass basis with an average of 30%, which means each tree can yield approximately 18.4 kg of oil, thus resulting in about 4800 kg of oil per hectare per year [13].

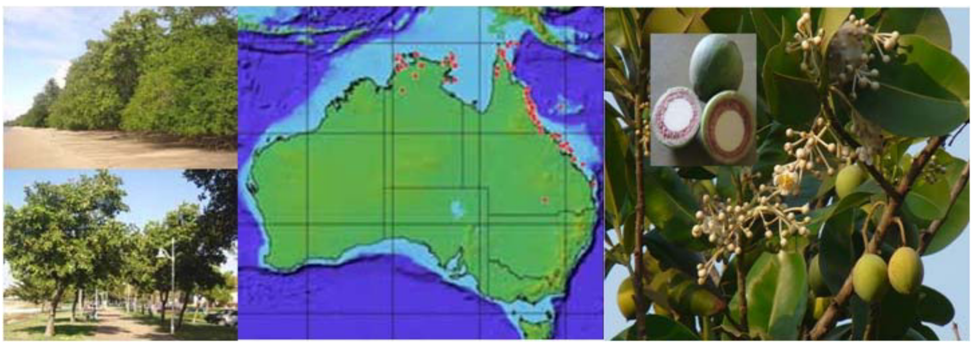

Fig. 1. *Calophyllum Inophyllum* fruit, growing along a beach front, and in a park and its distribution in Australia

3. Seed preparation

Approximately 140 kg of seeds had been procured from Australian native plant seed suppliers. These seeds were in a ground-dried state (i.e. had been on the ground for some time prior to collection). In this state the flesh was not present with the endocarp being the outermost layer. Furthermore, the kernels had shrunk somewhat from their fresh state and so a rattle could be heard when seeds were shaken.

3.1. Kernel extraction

Preparing the seeds for oil extraction involved first removing the outer layers to expose the kernel. It was necessary to crack the seeds open in order to obtain kernels for further preparation and processing. The seed cracking process used two methods: stompers and mallets. For the stomper, a large number of seeds were placed on the ground and worked until a number had been cracked, then the kernels and the waste husk were removed. For the mallet, operators placed a handful of seeds on the table surface and cracked them individually, before removing the kernels and the waste husk (Figure 2).

For the complete 140 kg of procured product approximately 51 kg of usable kernels were obtained. This translates to approximately 36% of the total procured mass being usable kernels. Small quantities of kernels had been used for some preliminary testing, so the as-cracked figure was slightly higher than 51 kg, but certainly not 60 kg as was predicted using the small-scale testing result. From this result it was concluded that the small scale test was simply not truly representative. One possible reason for this could be the location in the seed pile that the sample was taken from. It was also found that rubber-headed mallets were preferred less than wooden or steel-headed mallets, as they tended to rebound excessively. Using mallets meant that seeds were cracked either individually or only several at a time, whereas the stomper was capable of cracking numerous seeds at a time. However, due to the variability in size of the seeds, the efficacy of the stomper was reduced as it only struck the largest seeds with each blow. Overall, kernel extraction was found to be a time-consuming and labour-intensive process. It has been estimated that each method was roughly equal in cracking rate, at approximately 2–3 kg of seeds cracked per operator per hour; however cracking process may be automatable to increase the cracking rate.

Fig. 2. Beauty Leaf seed cracking area

3.2. Kernel drying

Experiment has been conducted to investigate the optimum moisture content of the seed kernel for high oil yield. Additionally, mulched and unmulched kernels were also investigated to determine the effect of mulching on the rate of drying. The drying process was conducted using a laboratory scale Clayson electric oven with temperature controller, as shown in Figure 4. Kernels were placed in the foil trays; generally 2 kg per tray to ensure the product was spread adequately for uniform drying in an oven (Figure 3). The drying progress was checked by weighing the trays several times daily when possible, and the mulched samples were also stirred at these times to provide aeration for uniform drying. When the samples had reached their desired dryness, they were immediately removed, bagged and placed in a refrigerated store room. Both mulched and unmulched kernels were dried at 50° C and 70° C to investigate the effect of mulching on drying rate and also temperature on drying rate. One sample has been dried for as long as possible to determine the absolute moisture content of the kernels. The kernel sample were obtained with about 9.5%, 15%, 20%, 25%, and 27% moisture content which were used to investigate the effect of moisture content on bio-oil production.

Fig. 3. Drying oven (left) and kernel trays inside (right)

For the drying rate comparison, the percentage dried was plotted against the drying time for both the mulched and unmulched samples, as shown in Figure 4. The result showed that the drying rate is higher for mulched samples. Drying of unmulched samples was roughly linear compared with mulched samples. The mulched sample also exhibited linearity although with a steeper gradient of just under 30% dryness achieved, at which point it began to plateau to a maximum of approximately 32%. For the same drying time (120 hours) the mulched sample was dried by approximately 32% while the unmulched sample was only dried by approximately 19%. The 70°C unmulched sample can be seen to dry at a higher rate compared with the 50°C unmulched sample. Again, these results are expected as drying a similarly prepared product at a higher temperature results in a faster drying rate. Interestingly, the drying characteristics are similar for the 70°C unmulched sample and the 50°C mulched sample. Therefore these results illustrated that kernel preparation could be an important factor in preparing efficient drying. Furthermore, it was noted that the maximum dryness achieved was approximately 32% which was adopted to be the absolute moisture content of the kernels. But this may vary depending on season session, location and maturity of the seeds.

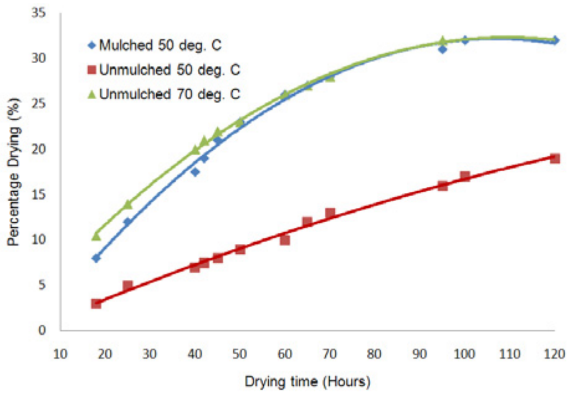

Fig. 4. Beauty Leaf seed kernel drying rate at 50 °C (mulched and unmulched) and 70 °C (ummulched)

4. **Oil extraction**

Two methods have been used for extracting oil from the prepared seed kernel. These are mechanical oil extraction using an electric powered screw press, and chemical oil extraction using n-hexane as a solvent. In both cases, experiments were conducted to find out the optimised moisture content for high oil yield.

4.1. Mechanical oil extraction

The mechanical oil extraction and experiment were conducted using a Mini 40 screw press at CPWS, Central Queensland University (CQU). As the screw press (Figure 5a) used in this study was not designed for Beauty Leaf seeds, it was realised that using this press for the oil extraction not only would this be challenging, but would require a degree of experimentation to adjust pressure and speed. Some modification also has been done to control proper operation. For example, glad wrap was used around the machine to capture any spilt material for the specific purpose of keeping mass control as precise as possible in order to give the most valid oil yield results.

(a) (b)

Fig. 5. (a) Mechanical oil extraction through a screw press (b) Chemical oil extraction

Beauty Leaf kernel was found to be very difficult to process using the screw press. Two operators were required to constantly attend to the machine and the rate of oil production was very low, typically taking over an hour to process just one sample. However, there was a noticeable increase in workability; as the samples became drier; oil was extracted much earlier so the extraction process was less laborious. The most consistently high oil yields were produced with 15% absolute moisture content, as shown in Figure 7. However, further improvement of Beauty Leaf oil extraction using the screw press is possible by optimising key design parameters of the machine including pressure, compression ratio, speed, and hot pressing.

4.1. *Chemical oil extraction*

For chemical oil extraction, dried seed kernels samples were ground using the blender and coffee grinder to obtain a fine consistency to maximise particle surface area. The ground kernels were put into conical flasks in which hexane were added at a ratio of 2:1 (mL hexane: grams kernel). The mixture was given an initial stir to ensure that all kernels were wetted with hexane. Conical flask openings were covered with aluminium foil and placed on the orbital mixer under the fume hood for safety reasons, and the samples were left to run for at least eight hours. Then the hexane/oil mixtures were collected, filtered and decanted into aluminium foil containers for solvent evaporation, and placed under the fume hood (Fig. 5b) for eight to 10 hours. Hexane was again added to the conical flask of kernels, but at a ratio of 1:1 for the second extraction, and a similar procedure was followed for recovery of the oil. When it was determined that the hexane had been fully evaporated, the oil was transferred into containers for analysis.

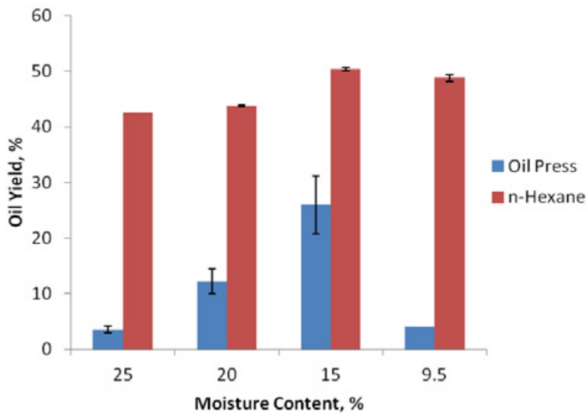

Fig. 6. Oil yield against absolute moisture content for chemically and mechanically processed samples

In terms of oil yield from the two extraction methods, hexane oil extraction was vastly more successful. For the test sample extractions (referring to Fig. 6), the samples with 15% absolute moisture content produced the highest oil yields, averaging approximately 51% (with respect to input dry mass). The difference was only slight between samples with an absolute moisture content of between 10% and 15%. However, the biggest obstacle for the hexane extraction method is the cost of the solvent. Therefore, is it strongly recommended to use the n-hexane recovery system to reduce the production cost of bio-oil production through the chemical method.

Table 1. Advantages and disadvantages of the two extraction methods

Mechanical Extraction		Chemical Extraction	
Advantages	***Disadvantages***	***Advantages***	***Disadvantages***
• Virgin oil is more sought after • No potential for solvent contamination • Relatively inexpensive after initial capital costs • Minor consumables cost	• Generally ineffective for processing Beauty Leaf • Time and labour intensive • Relatively low oil yields • Operators require experience to achieve best results • High dependence on kernel moisture content	• Repeatable and reproducible results and process • High oil yields • Relatively simple and quick • Hexane can be recovered and reused, reducing cost significantly	• Less sought after than virgin oil • Potential for solvent contamination • Safety issues and environmental concerns regarding the use of hexane • Very costly if the hexane cannot be recovered

5. **Comparison of oil extraction methods**

The results from the mechanical and chemical extraction methods clearly indicate that the hexane method is superior in terms of producing higher oil yields. It was also observed that the chemical method is more repeatable, relative ease of preparation and no requirement for extensive training. However, seed preparation has a significant impact on oil yields especially for the screw press extraction method. Kernels prepared to 15% moisture content provided the highest oil yields for both extraction methods. Mechanical extraction using the screw press can produce oil from appropriately prepared product, but overall this method is ineffective, with relatively low yields for a great deal of effort. Chemical extraction using hexane as a solvent was found to be very effective, but due to a limited supply of hexane and the lack of a hexane recovery system, it was not possible to take full advantage of the effectiveness of the method. Therefore, it is recommended to use chemical oil extraction with the hexane recovery system to reduce production cost of Beauty Leaf biodiesel. Moreover, as evidenced by the bulk extraction results, the hexane method proved to be a vastly more time and labour efficient process than mechanical extraction in the given circumstances. The advantages and disadvantages oil extraction methods observed in this study are summarised in Table 1.

6. **Conclusion**

Seed processing, drying and oil extraction methods a significant impact on oil yields and the success of Beauty Leaf as future generation biodiesel feedscock. Drying the seed kernel to optimum moisture content was found to be crucial to the success of both mechanical extraction and hexane extraction. Kernels prepared with 15% moisture content provided the highest oil yields for both extraction methods. Mechanical extraction using the screw press is ineffective, with relatively low yields for a great deal of effort. Chemical extraction using hexane as a solvent was found to be very effective, however due to a limited supply of hexane and the lack of a hexane recovery system, it was not possible to take full advantage of the effectiveness of the method. Therefore it is recommended to use chemical oil extraction with hexane recovery system to reduce the production cost of Beauty Leaf biodiesel. Though less crucial than hexane recovery, it is noted that kernel preparation was the most time consuming element of the hexane extraction process experienced during the project. Therefore, a potential area for improvement or a possible future project would be to investigate streamlining/automating and up-scaling the kernel preparation process.

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